Action of Ammonium Sulfate Solution on Dry Milk Powders

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Abstract

Milk powders dispersed in concentrated solution of ammonium sulfate maintain their structural configurations in spite of loss of lactose and calcium. In 95% saturated ammonium sulfate solutions, protein is not removed but lactose and calcium are rapidly and completely removed. In 50% saturated ammonium sulfate, some whey proteins were also removed without affecting the particles physical appearance. Some fragmentation occurred in all foam spray-dried powders probably from coalescence of interior gas bubbles in these less dense particles. Agglomerates of instantized skimmilk powder were broken down to their fundamental particles by the ammonium sulfate solutions, appearing thereafter much the same as noninstantized skimmilk particles. These phenomena indicate that, in the terminal stages of drying, proteins may interact to form a continuous phase in spray-dried milk and that lactose may be the bridging material in the formation of powder particle aggregates during instantizing.

Introduction

Growing concern about the intolerance of certain social groups to high lactose in their diets (1, 2, 6, 8) has renewed interest in low-lactose dairy product manufacture.

Early work by Leviton (7) demonstrated that lactose glass can be selectively leached from milk powder by aqueous solutions of methanol. Therefore, we decided to further investigate the selective solvation of milk powder constituents, primarily to gather fundamental information relative to milk fractionation and secondarily to study the factors responsible for milk powder particle structure. This paper reports results obtained by leaching milk powders with ammonium sulfate solutions.

Materials and Methods

Reagent grade chemicals were used throughout our work. The concentrated ammonium sulfate solution was made up to 95% saturation by addition of sufficient saturated solution to 5 ml of distilled water to make 100 ml of solution. The foam spray-dried whole milk and skimmilk powders were produced in our laboratory by conventional techniques (4). Samples of commercial milk powders were purchased in local supermarkets.

Photomicrographs of the milk powder particles were taken with a Polaroid attachment on a Bausch and Lomb Triocular Dynazoom microscope.[1] Calcium analyses on the powders and the ammonium sulfate filtrates were performed by atomic absorption with lanthanum added to the analytical solutions to minimize effect of the various interfering ions. Standard solutions were prepared to contain sulfate equivalent to that of the unknown in the analysis of filtrates. Lactose analysis was performed by the Munson-Walker method as modified by Fox et al. (3).

Five-gram samples of the powders were weighed into 200 ml Erlenmeyer flasks. Then 100 ml of concentrated ammonium sulfate solution were added and the flasks were quickly stoppered and agitation begun. For the samples to be photographed, agitation was continued for 30 min and then a deep well microscope slide was filled with suspension and sealed with a cover slip to prevent evaporation and subsequent crystallization of ammonium sulfate. The samples to be analyzed for lactose and calcium were agitated for varying times, then filtered through coarse filter paper with a Büchner funnel. Leaching was timed from the middle of the sulfate immersion to the middle of the filtration step.

Results

The appearance of the milk powders before and after immersion in ammonium sulfate solutions is shown in Figures 1 to 6. Immersion in ammonium sulfate caused little change in structure of foam spray dried whole milk (Fig. 1, 2). The agglomerates of instantized skimmilk powder, however, are broken down to their fundamental particles (Fig. 3, 4).

[1] Mention of brand or firm names does not constitute an endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.

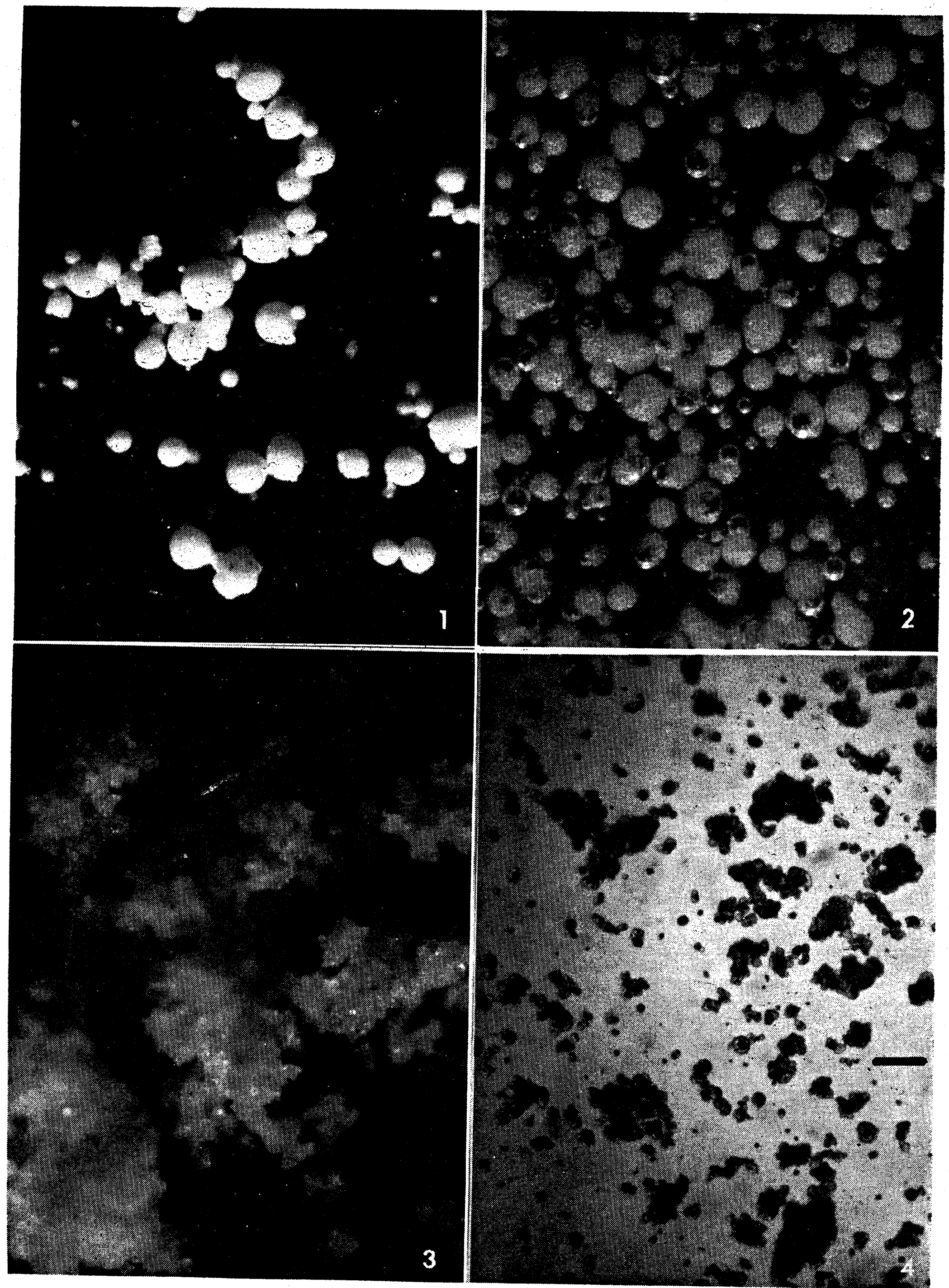


FIG. 1. Foam spray-dried whole milk particles in air.

FIG. 2. Foam spray-dried milk particles in ammonium sulfate solution.

FIG. 3. Instantized skimmilk powder in air.

FIG. 4. Instantized skimmilk powder in ammonium sulfate solution. Bar represents 200 μ and applies to Figures 1 to 4.

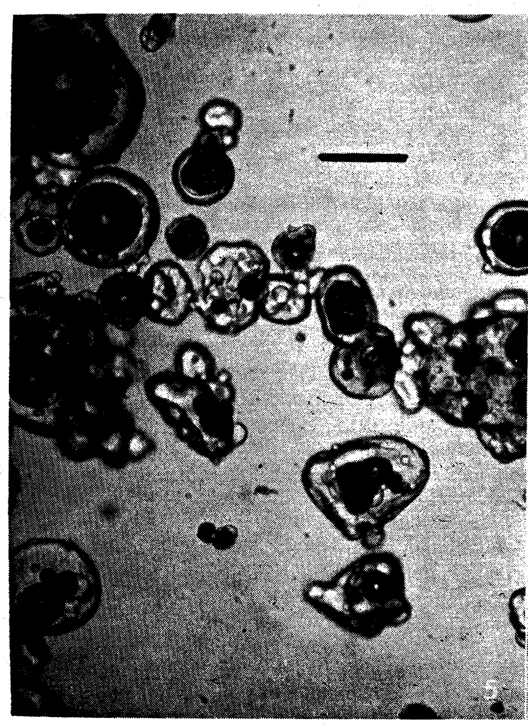
Figures 5 and 6 show, at higher magnification, that there is little, if any, difference between instantized and non-instantized skimmilk powder after immersion in ammonium sulfate solutions. Figures 7 and 8 show that both lactose and calcium were rapidly leached out of the powders by ammonium sulfate solutions.

Discussion

The primary conclusion from the foregoing results is that near saturated ammonium sulfate solutions cause no gross change in the physical appearance of the milk powder particles while completely removing lactose and calcium. Proteins were not leached out under these conditions as determined by disc electrophoresis. Half saturated solutions of ammonium sulfate yielded suspensions with the same appearance as Figures 1 to 6 but with some leaching of whey proteins. No quantitative estimates of this loss were made.

Concentrated ammonium sulfate solutions leached out a component which adsorbed light at 280 nm and proved to be dialyzable. A suspension of 10 g skimmilk powder in 100 ml saturated ammonium sulfate yielded a solution with absorbance of 5.5 (read on a diluted sample which was then corrected for dilution) dialysis against cold running tap water. This which was reduced to 0.05 absorbance by component may be the non-protein nitrogen fraction of milk since lactose and milk salts (as simulated by Jenness and Koops salt) do not exhibit absorption at 280 nm.

The preservation of the particles' appearance in ammonium sulfate may indicate that the protein therein may be forming a continuous phase. The rapidity of leaching of lactose and calcium indicates that the protein phase must be extremely porous. The per cent calcium leached out was computed from total calcium in the powders. No attempt was made to distinguish between the free, bound, or colloidal calcium of fluid milk. It can be postulated that during the terminal stages of drying, a protein network or sponge might be formed through unfolding of the casein micelle or through bridging of micelles by whey protein. An alternative model of a set of interacting spheres would require the casein micelle to be a porous body incorporating a considerable portion of the remaining constituents of milk. Packing of such polydisperse spheres can yield void space as low as 10 to 20% with 50% voids being reached only in a highly ordered array (5). Considering that casein comprises



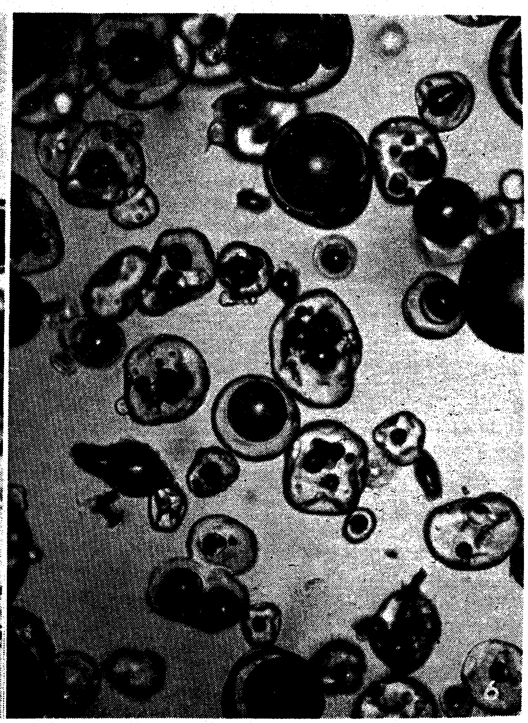


FIG. 5. Instantized skimmilk powder. Bar represents 50 μ and applies to Figures 5 and 6.

FIG. 6. Regular spray-dried skimmilk powder in ammonium sulfate solution.

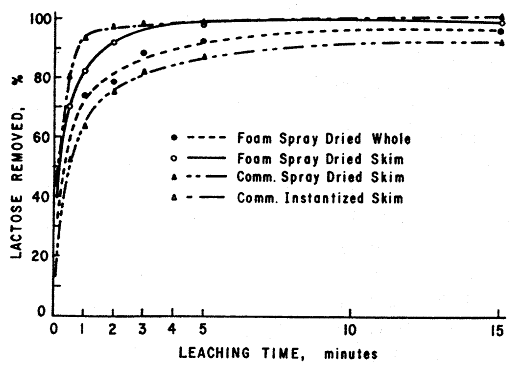


FIG. 7. Removal of lactose from milk powders by ammonium sulfate solutions.

about 20% of the solids of milk and choosing reasonable values for the densities of the solid components, it can be seen that no close packed array of pure casein spheres will yield sufficient voids to contain the remaining components of milk.

Attempts were made to determine the effect of the ammonium sulfate solutions on the particle size of the various powders utilizing an image splitting eyepiece. Individual particles were measured before and after immersion in ammonium sulfate solution. The most notable observation was a gradual shrinking of some of the particles, probably due to the effects of the concentrated salt solution. Evidence for a lactose coat by way of rapid initial shrinking was not found. It must be pointed out, however, that a measurable volume of material can be contained in a thin outer shell. For example, removing the outer 10% of a particle's volume will reduce the diameter only 3.4%. The uncertainty in our diameter measurements amounted to 1 to 2% during the first minute of immersion, hence a small coating of lactose would have gone undetected.

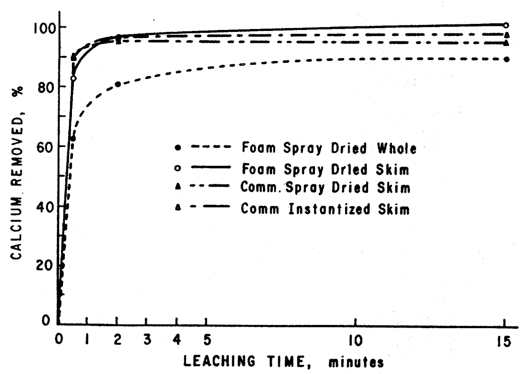


FIG. 8. Removal of calcium from milk powders by ammonium sulfate solutions.

The complete breakdown of aggregates of instantized skimmilk into their fundamental particles in ammonium sulfate solution strongly implicates lactose as the binding agent. The instantizing process apparently redissolved some small amount of lactose at the particle surface and allowed lactose bridge formation on intraparticle contact.

Speculation as to the effect of fat, especially of free fat, on leaching of milk components is tempting but not warranted on the basis of the small differences observed among the powders studied.

From the results we have concluded that suitable salt solutions may eventually be found that can be utilized to reduce the lactose content of milk powder and possibly provide an inexpensive method for fractionating cheese wheys. It is also obvious that milk powder structure needs further examination by electron microscopy of thin sections taken therefrom.

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